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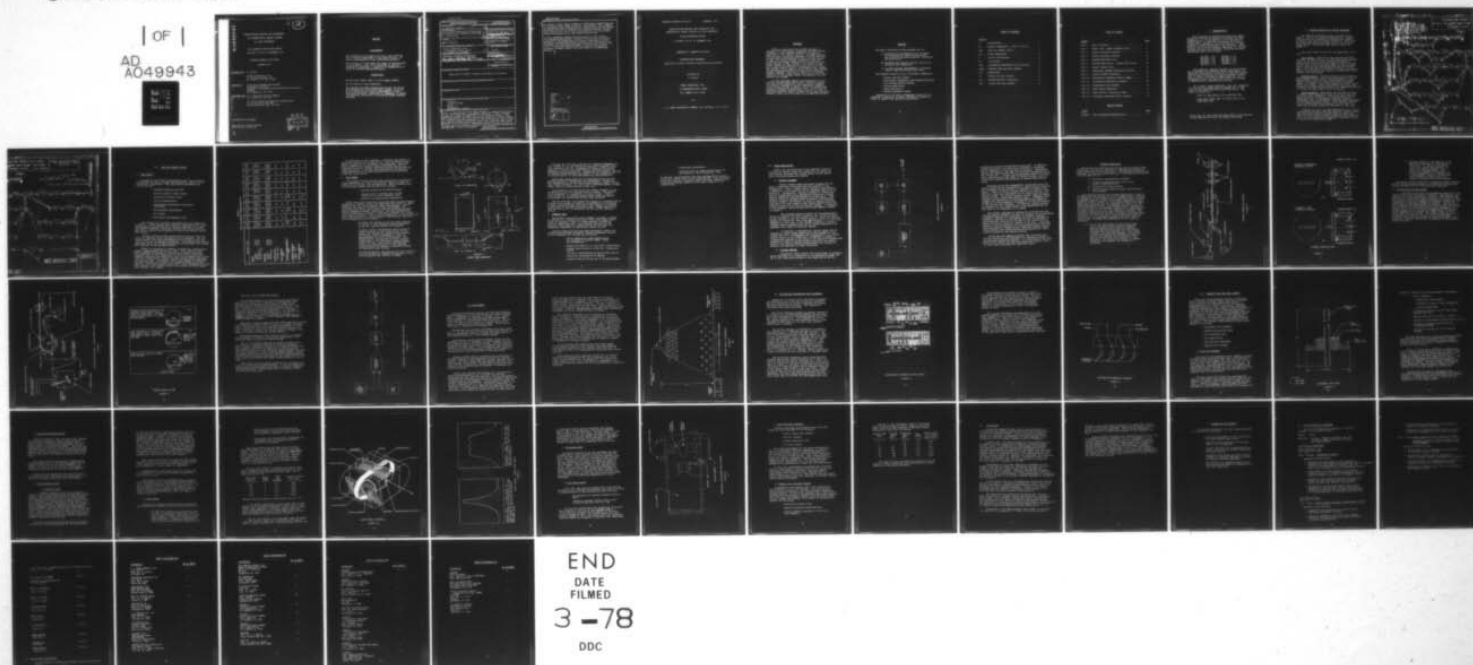
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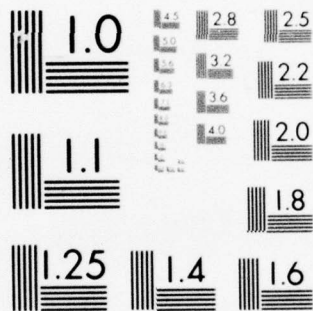
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MANUFACTURING METHODS AND TECHNOLOGY
FOR HERMETICALLY SEALED LITHIUM
SO₂ CELL BATTERIES

2nd QUARTERLY REPORT FOR PERIOD
1 OCTOBER 1976 TO 31 DECEMBER 1976

CONTRACT #DAAB07-76-C-0042

JANUARY 1977

PREPARED BY: T. Watson

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Mt. Vernon, New York 10550

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an effort to meet and/or exceed all performance specifications at minimum current density levels. Non-hermetic cell samples are presently being fabricated and tested under various thermal discharge profiles to simulate the proposed hermetic designs. Such results will aid in the final selection of the optimal design configuration for the hermetic prototypes.

Initial equipment design and fabrication of core winding, cathode/anode manufacture, hermetic closure and electrolyte preparation and dispensing is presently underway to permit subsequent integration within an operational production line. Interface with automated equipment manufacturers continue in order to fabricate specific machine elements and procure necessary hardware within the time frame of the PERT/TIME Network.

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TECHNICAL REPORT 0042-Q-02

JANUARY, 1977

MANUFACTURING METHODS AND TECHNOLOGY FOR
HERMETICALLY SEALED LITHIUM SO₂ CELL BATTERIES

SECOND QUARTERLY REPORT

1 OCTOBER 1976 TO 31 DECEMBER 1976

CONTRACT NO. DAAB07-76-C-0042

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Prepared by

T. Watson

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For

U. S. ARMY ELECTRONICS COMMAND, Fort Monmouth, N.J. 07703

ABSTRACT

Effort is continuing on the MM&T program to establish the fabrication techniques and requirements necessary to meet hardware production levels as specified in the subject contract. The PERT/TIME Network has been revised and expanded to define specific problem areas and establish a corrective action plan to meet the overall program objectives within the scheduled time frame. Hermetic cell and battery component design has been revised in an effort to meet and/or exceed all performance specifications at minimum current density levels. Non-hermetic cell samples are presently being fabricated and tested under various thermal discharge profiles to simulate the proposed hermetic designs. Such results will aid in the final selection of the optimal design configuration for the hermetic prototypes.

Initial equipment design and fabrication of core winding, cathode/anode manufacture, hermetic closure and electrolyte preparation and dispensing equipment is presently underway to permit subsequent integration within an operational production line. Interface with automated equipment manufacturers continue in order to fabricate specific machine elements and procure necessary hardware within the time frame of the PERT/TIME Network.

PURPOSE

The basic objectives of this program are to:

- a) establish the producibility of the specified hermetically sealed lithium cells and batteries by mass production techniques and facilities;
- b) establish and improve quality control surveillance and inspection;
- c) initiate process improvements to minimize overall fabrication costs and time.

The program consists of six (6) primary components:

- . Battery and Cell Design
- . Electrolyte Preparation and Dispensing System
- . Core Winding Machine Design
- . Cathode Manufacture
- . Anode Manufacture
- . Welding Equipment Design

Evaluation of the above independent tasks will be conducted in parallel to permit subsequent integration within an operational manufacturing process.

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I. INTRODUCTION

The Manufacturing Methods and Technology (MM&T) Project No. 2759371 to Establish Automatic Electrode Production for Lithium Hermetic Cells requires the establishment of production techniques for hermetic lithium cell components, cells and batteries to meet production levels delineated in the contract. Specifically, the following hermetic batteries will be manufactured utilizing the automatic electrode processes established under this program:

BA-5590 ()/U	BA-5574 ()/U
BA-5585 ()/U	BA-5841 ()/U
BA-5090 ()/U	BA-5100 ()/U
BA-5842 ()/U	BA-5567 ()/U
BA-5568 ()/U	BA-5598 ()/U

The production engineering goals of this program are to perform the necessary design, development, engineering, fabrication of special tooling and construction of test facilities and limited production equipment to obtain confirmatory sample approval; and to establish a pilot line and pilot run for the purpose of demonstrating a manufacturing process.

As a result, Power Conversion, Inc. will establish a Pilot Line and demonstrate the capability of this line with at least 20% of the Pilot Run units. The rates to be met are:

5,000 "D" Type Cells in an eight-hour day.

2,500 cells other than "D" Type* cells in an eight-hour day.

*Other than "D" type cells are those cells to be utilized in the fabrication of the deliverable batteries.

II. PROGRAM EVALUATION & REVIEW TECHNIQUE

The PERT/TIME Network has been revised and updated to reflect the present program status. Specific problem areas were defined and reviewed with respect to meeting the overall program objectives. Priorities have been restructured in order to meet the contract target dates. The revised PERT Chart is shown in Figure 1.

The major areas of revision are summarized as follows:

Cell Design - Additional time has been allocated to permit re-evaluation of the electrode design configuration in an effort to meet capacity and start-up requirements at minimum current density levels. In addition, lead time for the procurement of cell hardware and tooling has been determined from the various vendors. Such time must be included in order to accurately predict the scheduled time required to fabricate initial hermetic cell prototypes.

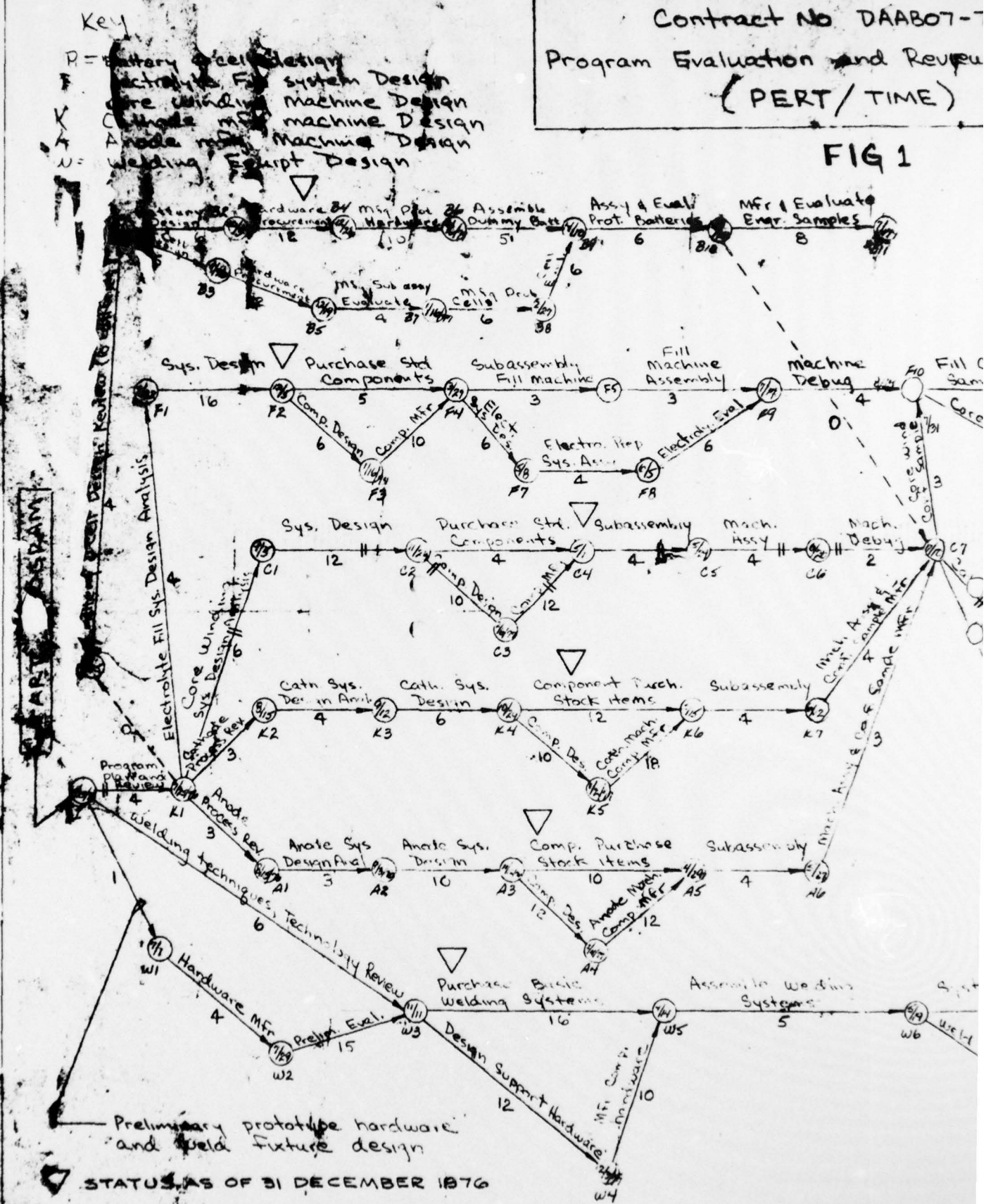
Battery Design - Various alternatives are presently being investigated to facilitate procurement of the required battery cases within a minimum period of time. Present time required to fabricate injection molded cases, for example, would delay completion of the initial battery prototypes. These alternatives are being evaluated and the PERT/TIME Network will be updated accordingly to reflect the most timely course of action.

Weld Equipment - Additional time has been allotted to permit a more complete evaluation of both laser and resistance weld techniques. Such evaluations include design selection of the optimal material handling techniques, cost of consumables, equipment and maintenance and anticipated product output. Since hermetic welding is a critical area of this program, we felt that insufficient time was previously allocated to this task.

Periodic review of the PERT/TIME Network will be continued during the next quarterly period in order to identify and define specific problem areas as they occur. Such action will permit timely corrective action and will avoid serious delays during component integration within an operational production system.

Contract No DAAB07-
Program Evaluation and Review
(PERT/TIME)

FIG 1



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Power Conversion Inc
Mt Vernon New York
July 1976

FIG 1

Mfr & Evaluate Ener. Samples 8

No. indicates avg. time in weeks

Double line indicates critical path

Total Critical Path Time (Avg) = 90 weeks

PROGRAM C

machine Debug 4

Fill Conf. Samples 3

Finish & Evaluation Function 12

Fill Pilot Cells 3

Finish Pilot Cells & Batteries 3

Evaluate 8

Core winding 4

Redesign upgrade 4

Comp. Mfr & Assembly 4

Component Evaluation 2

Pilot Run 2

Mach. Assy 4

Mach. Debug 2

Mach. Assy & Co & Sample Mfr 3

Main Debug 2

Component Evaluation 4

Redesign 5

Comp. Mfr & Assy 2

Pilot Run Anodes 2

Subassembly 4

Machine Winding Systems 5

System Debug 8

Upgrade Systems Re'd 12

Unit Evaluation 2

Unit Evaluation 2

Rev	Description
-1	Revised and updated 10/10

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Rev	Description
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III. CELL AND BATTERY DESIGN

A. CELL DESIGN

The specific electrode configurations have been re-defined in an effort to meet or exceed the performance, safety and environmental requirements. The basic design considerations were as follows:

- . Discharge Capacity and Rate
- . Minimal Current Density Levels
- . Operational Voltage Limits
- . Start-up Requirements
- . Dimensional Configuration and Weight Limitations
- . Environmental Requirements
- . Cell Safety
- . Fabrication and Material Costs

The revised cell electrode characteristics are summarized in Table 1. These designs were based upon maintaining the proper stoichiometric proportions of the active components, electrode utilization efficiency, available internal cell volume and minimum current density levels especially during high current pulsed duty cycles.

Although battery BA-5590 and BA-5842 utilize the same cell size, two separate design configurations are proposed. The discharge capacity and load requirements of the BA-5842 permit the use of a low rate electrode construction. This design incorporates components which are far more economical than the high rate counterpart required in the BA-5590 battery.

Effort is continuing to develop a bottom fill configuration using a flanged eyelet as the fill port. Such a design would permit greater utilization of internal cell volume; especially for cells used in the construction of the BA-5567 and BA-5568. Hermetic closure of the bottom fill port can be accomplished within .080 inch; significantly closer than the present design configuration of .185 inch. In addition, there is no danger of mechanical or thermal damage to the glass seal assembly during the closure process. This design permits the use of a flat reinforced top structure instead of the present concave shell design to further maximize internal cell volume.

TABLE 1
CELL ELECTRODE CHARACTERISTICS

BATTERY TYPE	5598	5100	5590	5842	5585	5567	5568	5841	5574	5090
<u>ANODE</u>										
Length (inch)	27.0	27.0	21.0	11.5	23.0	11.5	10.0	5.5	5.5	4.5
Width (inch)	1.375	.562	1.625	1.625	1.625	.200	.200	2.125	.750	.500
Thickness (inch)	.012	.012	.012	.019	.008	.012	.012	.010	.010	.010
Weight (gm)	3.9	1.8	3.6	3.1	2.6	.24	.21	1.0	.36	.2
<u>CATHODE</u>										
Length (inch)	28.0	28.0	22.0	12.0	24.0	12.0	10.5	6.0	6.0	5.0
Width (inch)	1.375	.562	1.625	1.625	1.625	.200	.200	2.125	.750	.500
Thickness (inch)	.033	.033	.033	.045	.020	.033	.033	.024	.024	.024
<u>ELECTROLYTE</u>										
Weight (gm)	34	20	34	34	15	4.5	3.5	8.0	4.0	2.5
<u>CELL WEIGHT</u> (gm)	90	37	85	85	58	12	12	23	12	7
<u>CURRENT REQUIREMENTS</u> (amps)	.95 .048	.093	3.0 .64 .05	.175	3.0 .64 .05	.056	.090	.12	.115	.013
<u>CATHODE SURFACE</u> AREA (cm ²)	497	226	461	252	503	31	27	165	58	32
<u>CURRENT DENSITY</u> LEVEL (ma/cm ²)	1.91 .10	.41	6.5 1.4 .11	.69	6.0 1.3 .10	1.81	3.33	.73	1.98	.41

An evaluation is also underway to determine the effects of various PCI electrolyte formulations on cell discharge performance and operation of the safety vent mechanism. Cells and control samples are currently being fabricated and will be subjected to various thermal discharge profiles during the next reporting period. Following data accumulation and analysis, a determination will be made regarding the final electrolyte recipe and constituents.

B. CELL SAFETY

Our primary concern has been the optimization of effective safety mechanisms to insure non-hazardous operation under all conditions of storage, use and operation. Basic considerations for such safety mechanisms include the following:

- . System Reliability and Effectiveness
- . Minimal Loss of Internal Cell Volume;
Especially for Small Diameter Cells
- . Economic and Reproducible Fabrication

Effort during this quarterly period has been directed towards optimizing a reliable safety vent mechanism which could be implemented on all cell sizes required under this program. Such a universal vent design simplifies tooling requirements and facilitates the design of automatic material handling equipment during subsequent assembly operations. The most promising design as illustrated in Figure 2 essentially consists of a coined cross section located in the can wall and parallel to the center line. The coined structure is rolled radially inward during fabrication which accomplishes the following:

- . Minimizes the bulging of the can outer diameter to conform to the required envelope dimensions during all required thermal environments.
- . Provides a pivot configuration which minimizes tensile loading of the coined surface during normal cell storage and operating environments. The flexure joint is designed to invert at approximately 200-210°F at which point, the coined section is subjected to direct tensile loading due to the electrolyte vapor pressure within the cell. Such action provides greater control and reproducibility of the venting characteristics and permits a graduated release of electrolyte upon venting.
- . Provides protection and isolation of vent structure during cell/battery assembly and handling to prevent accidental vent rupture or damage.

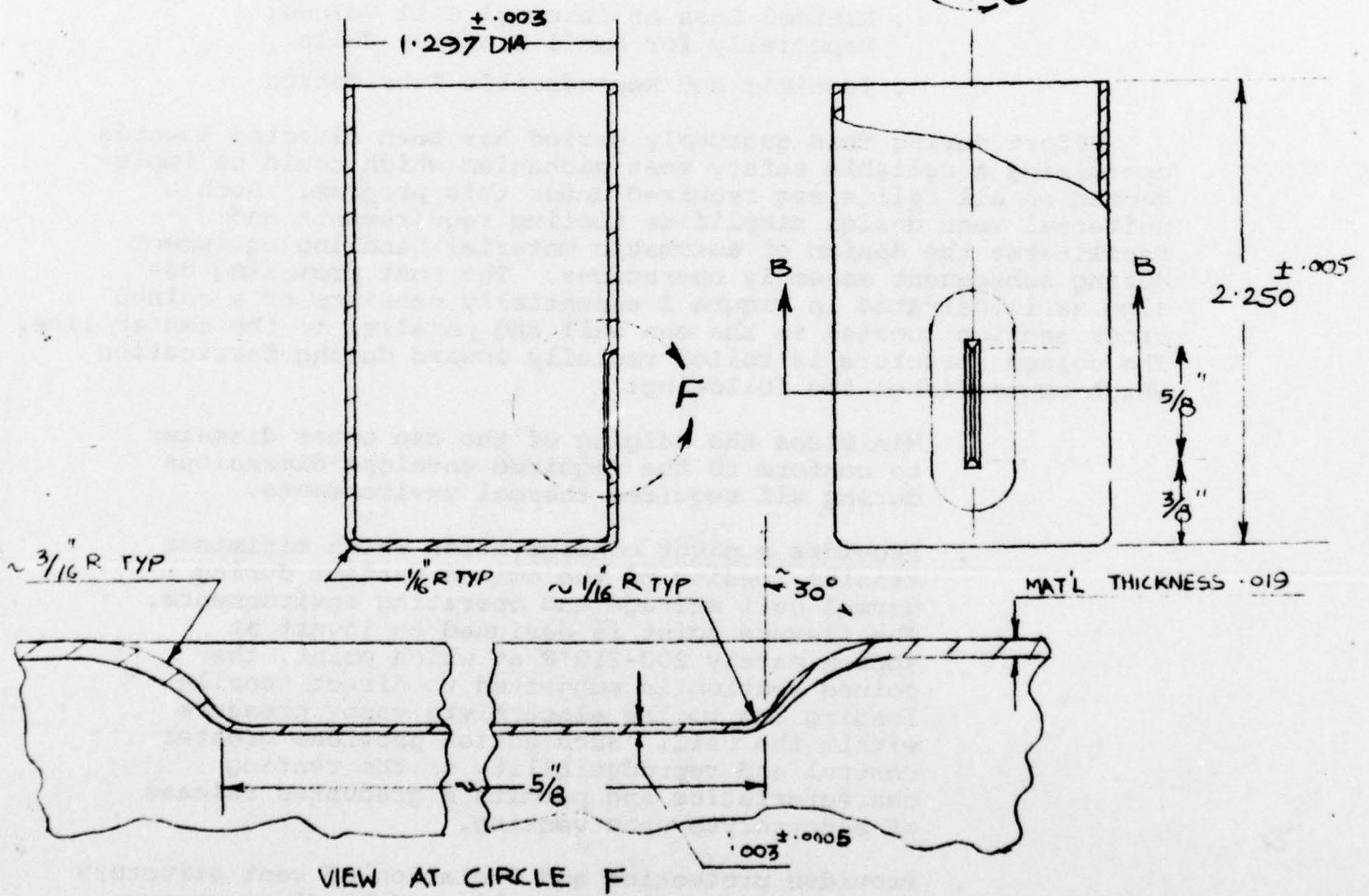
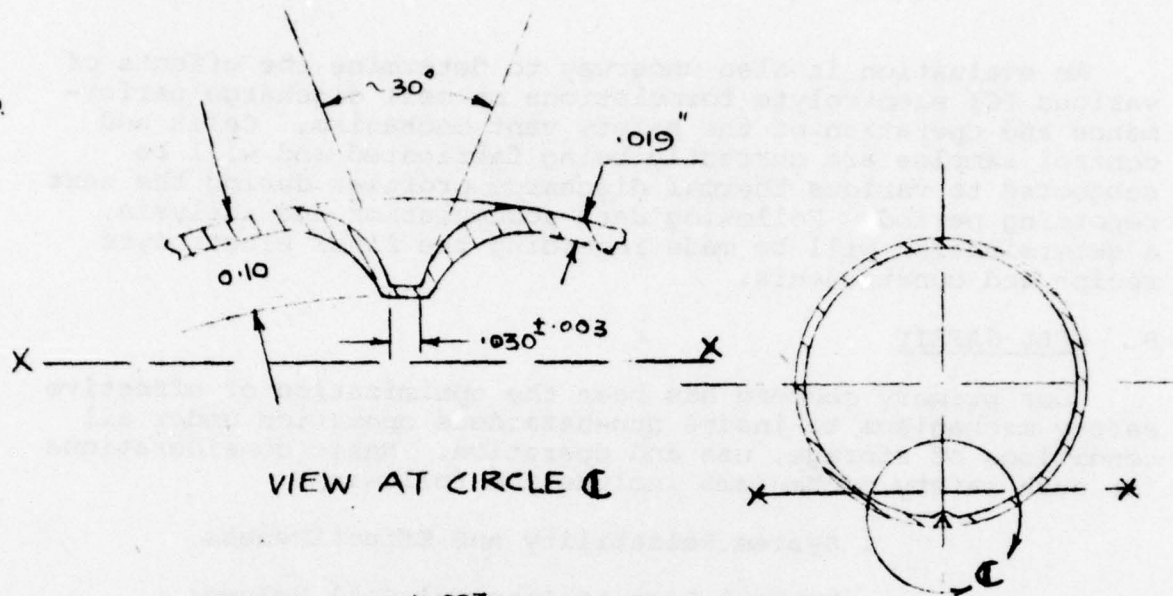


FIGURE 2
SAFETY VENT MECHANISM

The cans are initially annealed in a reducing atmosphere at 1750 -1800°F for 4.5 minutes to provide uniform material hardness throughout the can. Hardness uniformity is particularly important to control the coined section thickness during the subsequent forming operations. Evaluations are presently underway to quantitatively determine the effects of post annealing of the coined section on vent performance and reproducibility.

Preliminary abuse tests will be performed during the next quarterly period to demonstrate the performance, reliability and reproducibility of the vent during exposure to short circuit, high current discharge, hot plate tests, elevated thermal storage tests, hydraulic pressurization tests, etc.

Vent pressure is controlled by the overall geometry of the coined section and the relative state of anneal. The optimal venting pressure is considered to be 425 ± 25 psi. Such a value allows for an adequate safety margin above the maximum anticipated cell operational/storage vapor pressure.

The PCI side wall vent design will result in a minimal loss of internal cell volume; an important consideration in the design of small diameter/height cells since cell capacity is directly proportional to available internal volume. Space required for vent activation will be determined during the next quarterly period.

C. HERMETIC SEAL

The hermetic glass/metal seal assembly, as shown in Figure 12 consists of a tantalum tube and a cold rolled steel eyelet which are thermally fused to a glass preform to effect an hermetic compression seal. The tantalum tube acts as the positive interconnection terminal and as the fill port for subsequent dispensing of electrolyte within the cell.

Detailed specifications have been developed to define the manufacturing process and quality control acceptance criteria of this component and include the following:

- . Visual examination under magnification for evidence of surface cracks, voids and delamination.
- . Hermeticity Test @ 2.0×10^{-8} atm. cc/sec helium
- . Thermal Shock @ -65c to +125c for 3 continuous cycles.
- . Dielectric Withstanding Voltage @ 1500 volts AC
- . Insulation Resistance @ 50 megohms
- . Pressure Tests @ 500 psi for 30 seconds minimum

. Cleanliness Requirements

- . Ductility Test to assure proper pinch off
and closure of the tantalum fill tube.

In addition, specifications have been implemented to control the surface defect level of the tantalum tube. Such corrective action was required to prevent minute capillary leakage along longitudinal surface defects on the outer diameter of the tantalum tube.

IV. Anode Fabrication

Effort has continued during this quarterly period to finalize the design stages for integrating the anode tab cold welding technique and lithium rotary slitter design into a prototype production machine.

A. Lithium Transfer

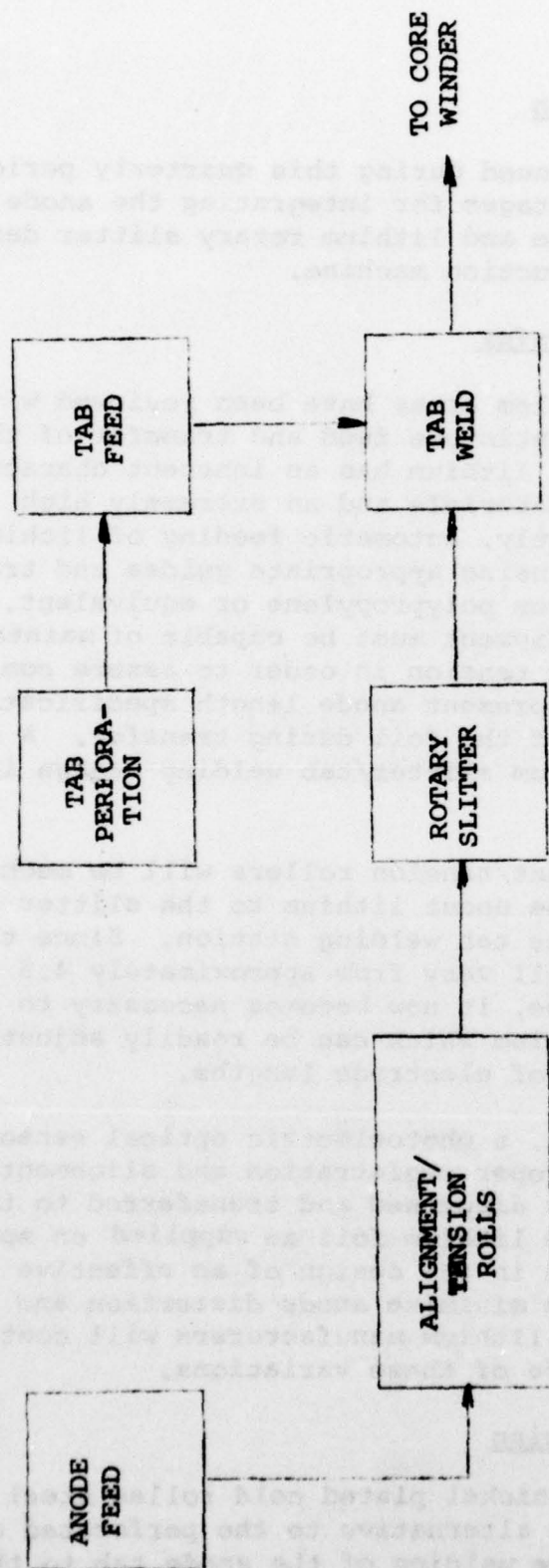
Several problem areas have been reviewed with respect to achieving continuous feed and transfer of the lithium foil. First, lithium has an inherent characteristic of adhering to most materials and an extremely high level of ductility. Consequently, automatic feeding of lithium foil must be accomplished using appropriate guides and transfer rollers fabricated from polypropylene or equivalent. Such material handling equipment must be capable of maintaining uniform alignment and tension in order to assure consistent cuts which will meet present anode length specifications and minimize distortion of the foil during transfer. A schematic of the proposed lithium slitter/tab welding design is shown in Figure 3.

The alignment/tension rollers will be mechanically driven to transfer the uncut lithium to the slitter and again during transfer to the tab welding station. Since the required lengths of lithium will vary from approximately 4.5 to 27.0 inch depending on cell type, it now becomes necessary to design a transfer guide mechanism which can be readily adjusted to accommodate this range of electrode lengths.

In addition, a photoelectric optical sensor may be required to insure proper registration and alignment of the lithium foil as it is dispensed and transferred to the slitter. Lateral runout of the lithium foil as supplied on spools is a significant problem in the design of an effective transfer/feed mechanism in order to minimize anode distortion and bending. Liaison with various lithium manufacturers will continue to minimize the magnitude of these variations.

B. Lithium Tabbings

Pre-pierced nickel plated cold rolled steel is presently being evaluated as an alternative to the perforated copper tab now in use. Resistance welding of the anode tab to the can



ANODE FABRICATION PROCESS

FIGURE 3

structure is greatly facilitated by using CRS. In addition, stress corrosion at the weld joint interface is minimized due to commonality of the tab and can material. Preliminary results now indicate that CRS will cold weld satisfactorily to the lithium foil. Voltage drop measurements across the welded joint interface have been taken at various current levels to verify weld integrity and establish a maximum specification value.

Prototype cells were subsequently constructed using the nickel plated CRS tab configuration to quantitatively evaluate cell discharge characteristics as compared to standard cell design. After an initial storage period of 16 hours at 130°F, both cell types were discharged at low, medium and high rate current drains. The results indicate similar capacity characteristics within normal distribution limits. Subsequent disassembly and examination of the discharged cells showed no evidence of electro-chemical corrosion at the CRS tab/lithium or CRS tab/can weld interface. In addition, surface contact and adhesion of the lithium anode to the perforated CRS tab was found to be acceptable in all cases.

The present technique of folding the electrode end over the pre-pierced conductor tab prior to staking is not considered a feasible approach for developing an automatic sequence. Therefore, staking of a pre-pierced tab will be performed at pre-determined intervals on one side of the lithium foil to enable continuous electrical tabbing of the foil. Continuous feeding of the tab material will be evaluated during the next reporting period to determine the trade-offs between feeding of pre-pierced tabs versus feeding of continuous foil which would be pierced at the staking station.

During the next reporting period, PCI will begin the initial fabrication and procurement of components needed for the prototype production machine. It is anticipated that this machine will achieve the specified production rate of 5,000 "D" size anode electrodes per eight-hour shift.

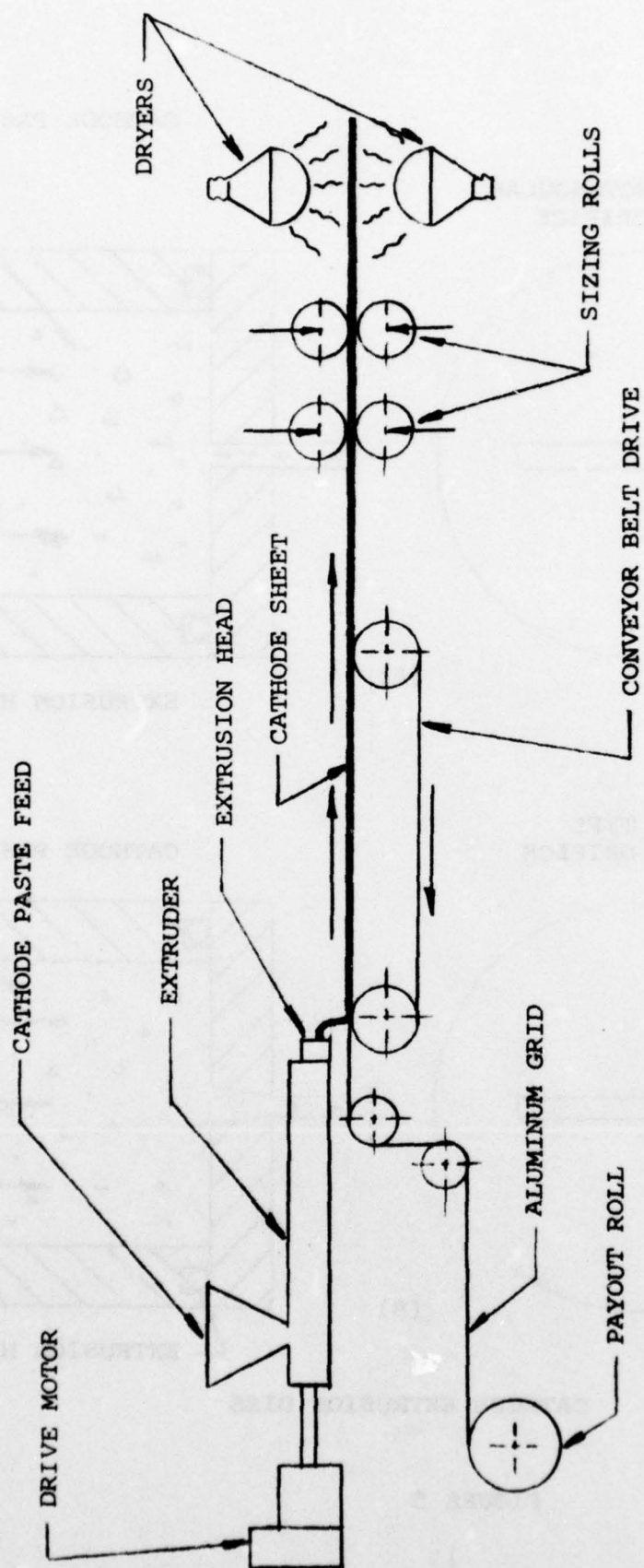
CATHODE FABRICATION

Work during this quarterly period has been directed towards developing a continuous technique for fabricating cathodes at rates consistent with program goals. Two potential manufacturing processes for fabricating cathodes were investigated concurrently with the objective of:

- a) Minimizing developmental problems;
- b) Providing maximum process control and product reproducibility;
- c) Maintaining product quality;
- d) Achieving the production goals required under this program.

One continuous process for cathode fabrication is that of extruding the raw cathode mix into an endless sheet, followed by lamination of this sheet onto an expanded screen conductor. A schematic drawing indicating the basic components for this system is shown in Figure 4. Preliminary bench scale experiments have indicated that this approach is viable, i.e. the PTFE/water/carbon mixture is extrudable and this extrusion can be pressed into an expanded aluminum grid. Specifically, extrusion trials at PCI for the formation of cathode sheets utilized a three-inch screw type Enterprise Extruder. Two different extrusion dies as shown in Figure 5 were fabricated and tried. It was found that a simple extrusion plate (b) works as well as a gradually tapered "fish-tail" type extrusion die. Problems were encountered in two areas of extrusion:

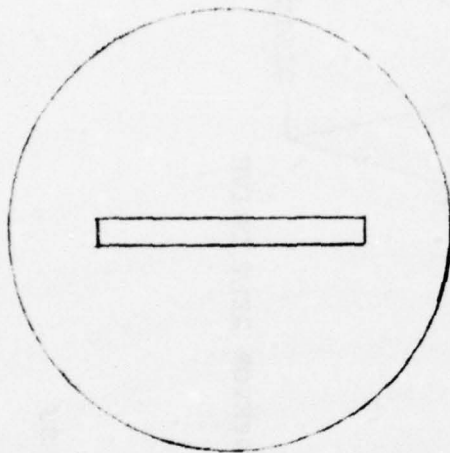
- 1) Due to the large amount of moisture present in the raw mix, during material compaction water is expelled and a moisture gradient develops across the extruder with the driest material near the discharge end. This caused a gradual slow down and stoppage of extrusion as the "wet" material at the feed end was unable to displace the "dry" material at the discharge. By reducing the amount of trapped moisture, this gradient can probably be minimized.



CATHODE EXTRUSION PROCESS

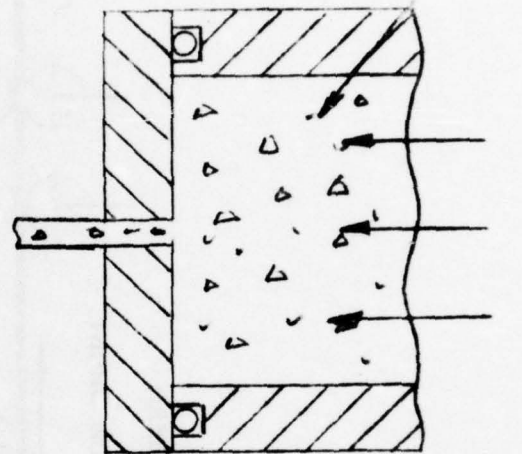
FIGURE 4

STANDARD RECTANGULAR
EXTRUSION ORIFICE



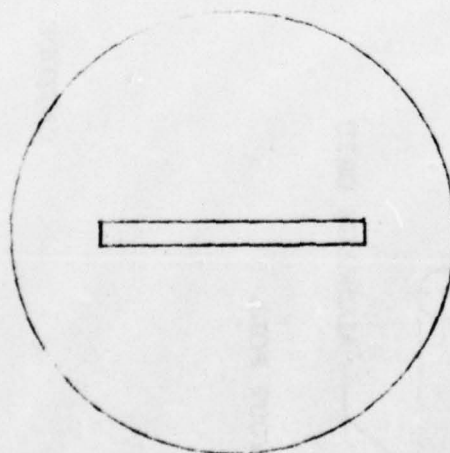
(A)

CATHODE PASTE



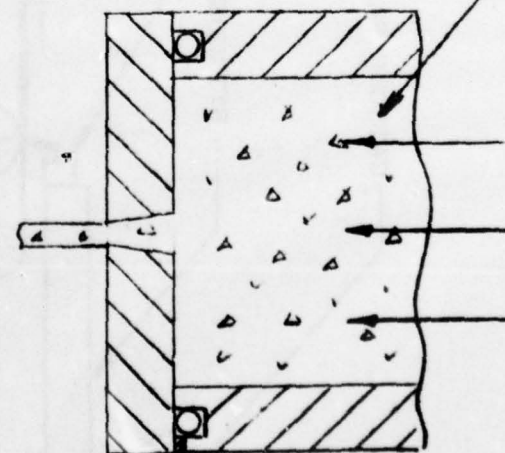
EXTRUSION HEAD

"FISHTAIL" TYPE
EXTRUSION ORIFICE



(B)

CATHODE PASTE



EXTRUSION HEAD

CATHODE EXTRUSION DIES

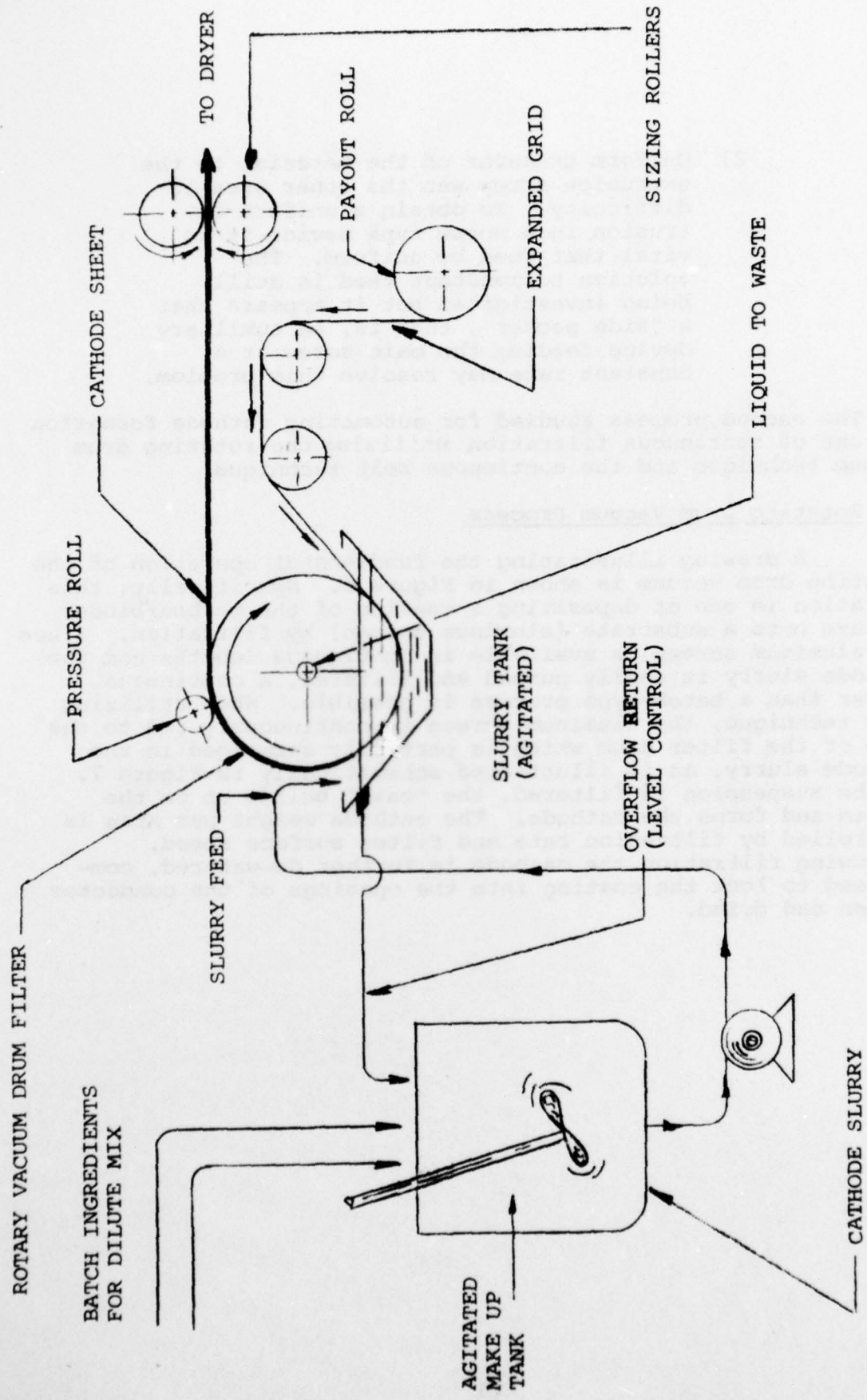
FIGURE 5

- 2) Uniform transfer of the material to the extrusion screw was the other area of difficulty. To obtain a uniform extrusion in a screw type device it is vital that feed be uniform. The solution to constant feed is still being investigated but it appears that a "side packer", that is, an auxiliary device feeding the main screw at a constant rate may resolve this problem.

The second process studied for automating cathode formation was one of continuous filtration utilizing the rotating drum vacuum technique and the continuous belt technique.

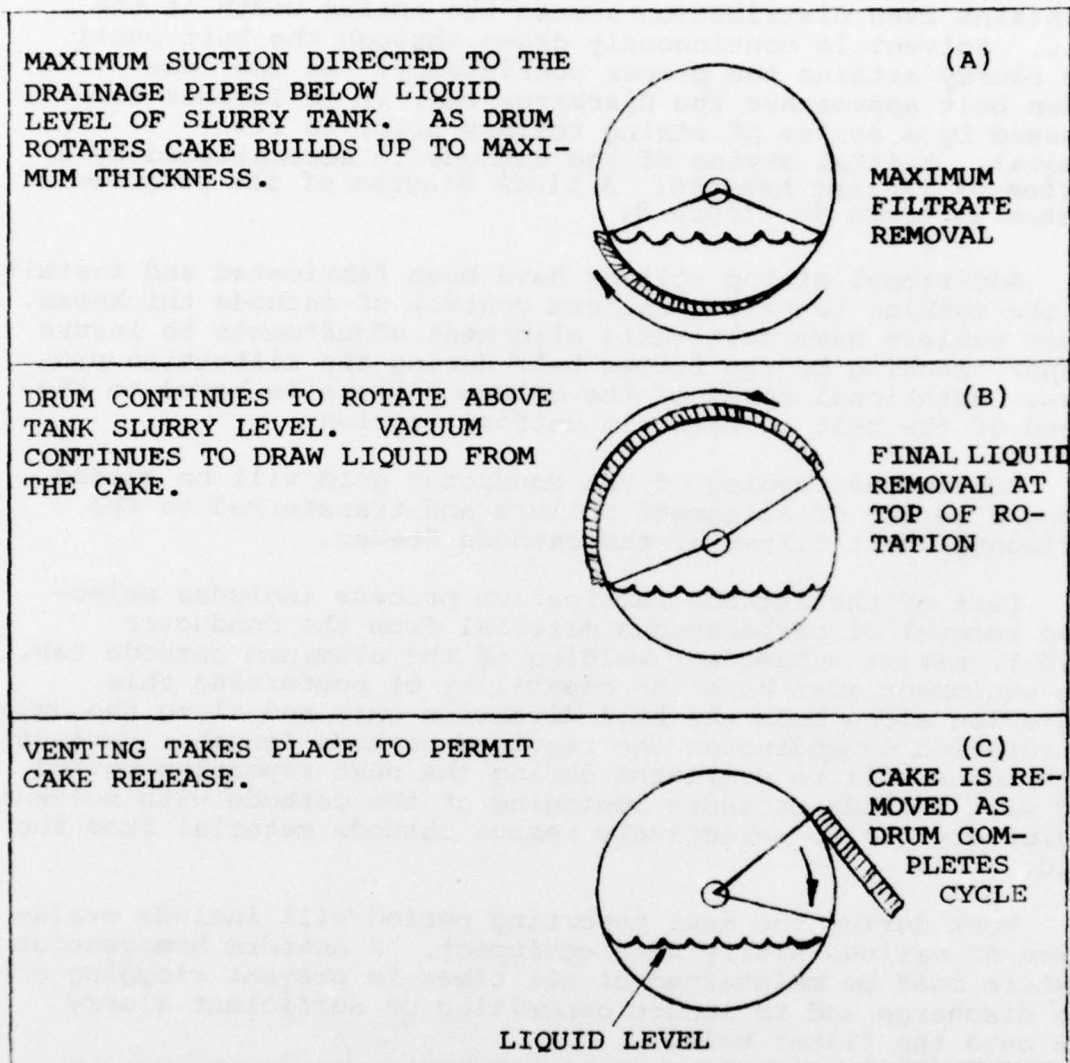
A. Rotating Drum Vacuum Process

A drawing illustrating the fundamental operation of the rotating drum vacuum is shown in Figure 6. Specifically, this operation is one of depositing a coating of the carbon/binder mixture onto a substrate (aluminum screen) by filtration. Since the aluminum screen is available in continuous lengths and the cathode slurry is easily pumped and filtered, a continuous rather than a batch-type process is possible. When utilizing this technique, the aluminum screen is continuously fed to the face of the filter drum which is partially submerged in the cathode slurry, as is illustrated schematically in Figure 7. As the suspension is filtered, the "cake" builds up on the screen and forms the cathode. The cathode weight per area is controlled by filtration rate and filter surface speed. Following filtration the cathode is further de-watered, compressed to lock the coating into the openings of the conductor screen and dried.



CATHODE FABRICATION - ROTARY DRUM FILTER

FIGURE 6



ROTARY VACUUM FILTER

FIGURE 7

D. CONTINUOUS BELT FILTRATION PROCESS

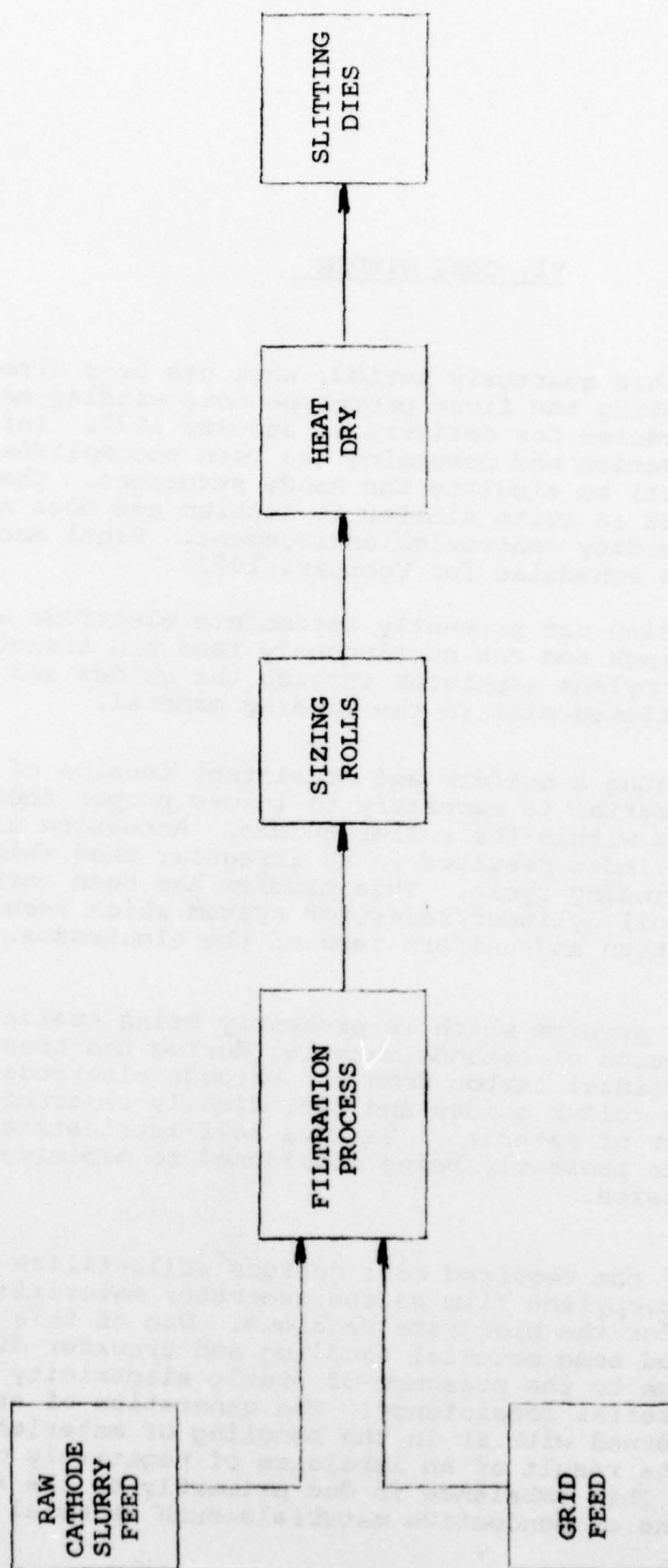
The selected approach for the first prototype machine essentially consists of a horizontal traveling belt that receives the raw cathode slurry through a feeder which maintains even distribution across the entire width of the belt. Solvent is continuously drawn through the belt until the slurry attains the proper consistency. As the cake laden belt approaches the discharge end, it is further compressed by a series of sizing rollers prior to its removal. Initial drying of the cathode is accomplished by a series of radiant heaters. A block diagram of the proposed system is shown in Figure 8.

Additional sizing rollers have been fabricated and installed on the machine to permit uniform control of cathode thickness. These rollers have multi-axis alignment adjustments to insure proper tracking of the filter belt during the filtration process. Rotational speed of the driven rollers is keyed to the speed of the belt to maintain uniform tension.

Continuous feeding of the conductor grid will be guided along a series of alignment rollers and transferred to the horizontal belt filter at the cathode feeder.

Part of the cathode fabrication process includes selective removal of carbonaceous material from the conductor grid to permit subsequent welding of the aluminum cathode tab. The equipment must have the capability of performing this operation along both the belt direction axis and along the axis of rotation depending on the required cathode length. Various techniques will be evaluated during the next reporting period and will include pressure impinging of the cathode with solvent and/or dry air to selectively remove cathode material from the grid.

Work during the next reporting period will include evaluation of various slurry feed equipment. A uniform homogeneous mixture must be maintained at all times to prevent clogging of the discharge and to insure depositing of sufficient slurry mix onto the filter belt.



CONTINUOUS CATHODE FABRICATION PROCESS

FIGURE 8

VI. CORE WINDER

During this quarterly period, work has been directed toward completing the first prototype core winding machine which is scheduled for delivery in January 1977. Initial machine evaluation and debugging has been accomplished using lead foil to simulate the anode structure. The ductility of lead is quite similar to lithium and does not require a humidity controlled environment. Final machine evaluation is scheduled for February 1977.

The machine can presently accomodate electrode widths up to 1 7/8 inch and can continuously feed and transfer non-woven polypropylene separator through the guides and into the pre-positioned slot in the winding mandrel.

Maintaining a uniform and consistent tension of the separator material is necessary to insure proper feed rate and alignment within the roller guides. Actuation using a pneumatic cylinder resulted in an irregular feed which disrupted the winding cycle. This problem has been corrected by using an oil cylinder/reservoir system which results in smooth actuation and uniform feed of the electrodes.

Another problem which is presently being evaluated is surface abrasion of cathode material during the transfer process. Residual carbon from the cathode electrode accumulates on the roller guides and periodically obstructs the free transfer of material. Various self-lubricating roller materials are presently being considered to minimize this surface abrasion.

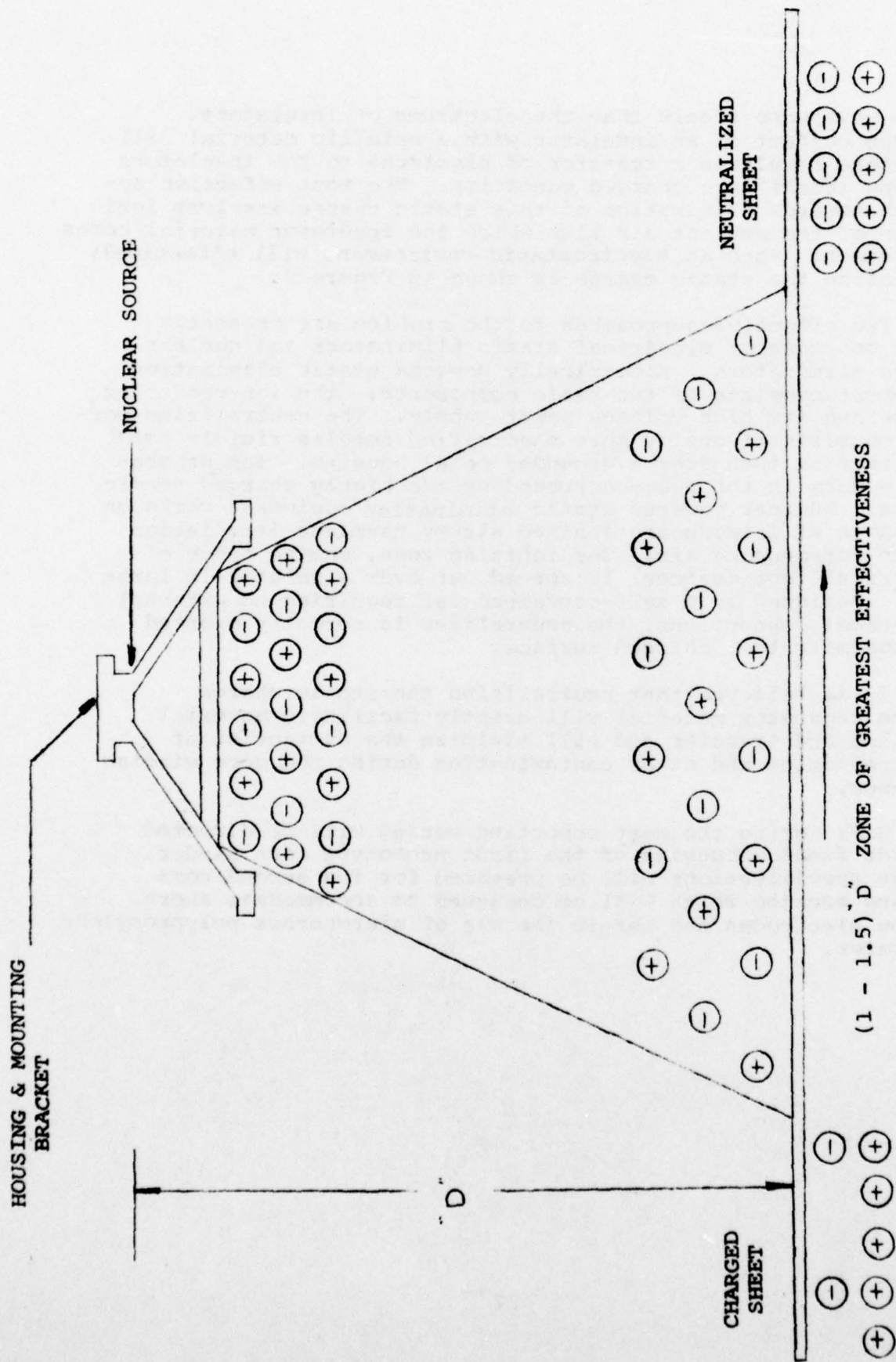
Some of the required cell designs will utilize micro-porous polypropylene film as the separator material; especially for the high rate versions. Use of this material has presented some material handling and transfer difficulties; primarily due to the presence of static electricity and the inherent material consistency. The generation of static, as we are concerned with it in the handling of materials, is basically the result of an imbalance of negatively charge electrons. This imbalance is due primarily to the fact that the electrons of conductive materials such as metal machine

parts move more freely than the electrons of insulators. Surface contact of an insulator with a metallic material will therefore result in a transfer of electrons to the insulators leaving itself in a charged condition. The most effective approach towards elimination of this static charge involves ionization of the ambient air with which the insulator material comes in contact. Such an electrostatic environment will effectively neutralize the static charge as shown in Figure 9.

Two effective approaches to the problem are presently being considered; electrical static eliminators and nuclear static eliminators. Electrically powered static elimination equipment consists of two basic components: the ion-producing source and its high voltage power supply. The neutralizing portion consists of one or more electrified needles rigidly held less than an inch from a grounded metal housing. Ion generation occurs in the space surrounding the highly charged needle points. Nuclear powered static elimination equipment works on the basis of introducing ionized air by harmless irradiation of the surrounding air. The ionizing zone, unlike those of electrical type devices, is spread out over a relatively large area. Designed in a self-contained bar requiring no external electrical connections, the neutralizer is normally mounted in proximity to a charged surface.

It is believed that neutralizing the static charge of the separator material will greatly facilitate material handling and transfer and will minimize the attraction of dust particles and other contamination during the core winding sequence.

Work during the next reporting period will be directed towards final debugging of the first prototype core winder. Design specifications will be prepared for the second core winding machine which will be designed to accommodate short, narrow electrodes and permit the use of microporous polypropylene separator.



NUCLEAR STATIC ELIMINATOR

FIGURE 9

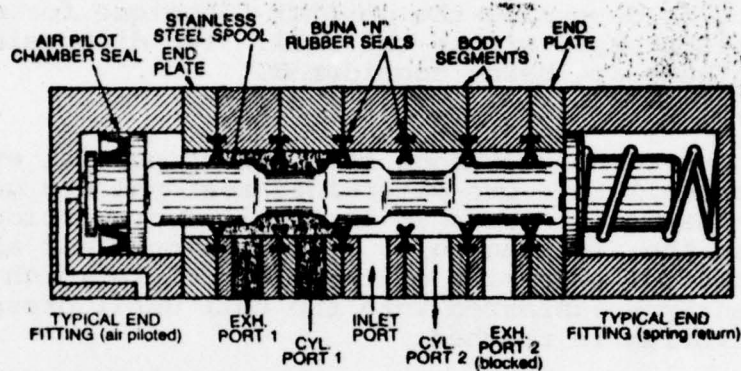
VII. ELECTROLYTE PREPARATION AND DISPENSING

Evaluation of various valve and metering dispensing systems has been continued during this quarterly period to determine the optimal technique for dispensing electrolyte within the cell. Two dispensing techniques are being considered.

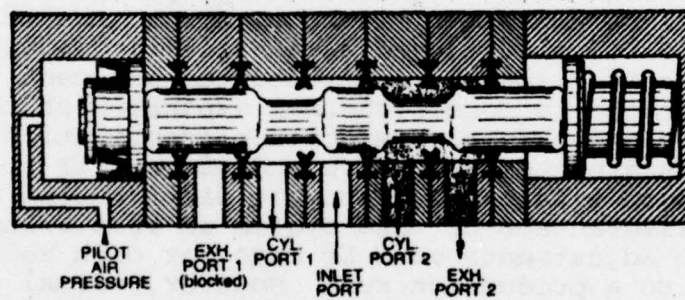
The first technique consists of partial evacuation of the cell to a pre-determined residual dry gas pressure that corresponds to the desired proportion of dry gas in the finished cell. This residual air allows for thermal expansion of the electrolyte which is subsequently transferred into the cell until pressure equilibrium is reached.

The second technique is essentially a volumetric fill system using a graduated syringe to meter a pre-determined amount of electrolyte into the cell. The cell is fully evacuated and a specific amount of electrolyte is dispensed. Five to ten per cent void space is allotted to permit thermal expansion of the electrolyte and prevent hydraulic pressurization. Cell weights are checked before and after fill to insure operation within an acceptable distribution curve. One disadvantage of this system is that volumetric set-up adjustments must be made for each cell size prior to a production run. However, normal electrolyte volume variations can be controlled to a minimum level.

The electrolyte dispensing valve system has been reviewed during this quarterly period in an effort to finalize the optimal design configuration. Such a system will consist of a series of 4-way shuttle valves as shown in Figure 10. The basic valve body contains stainless steel or aluminum spools which transverse from end to end within seals locked in the valve body. Because of the gradual transition between major to minor spool diameters, sealing is achieved with minimum friction and without the seals being squeezed across holes that could cause them to extrude, cut and prematurely wear.



Cross-section of typical 5-port
4-way Capsula valve in the unactuated
condition



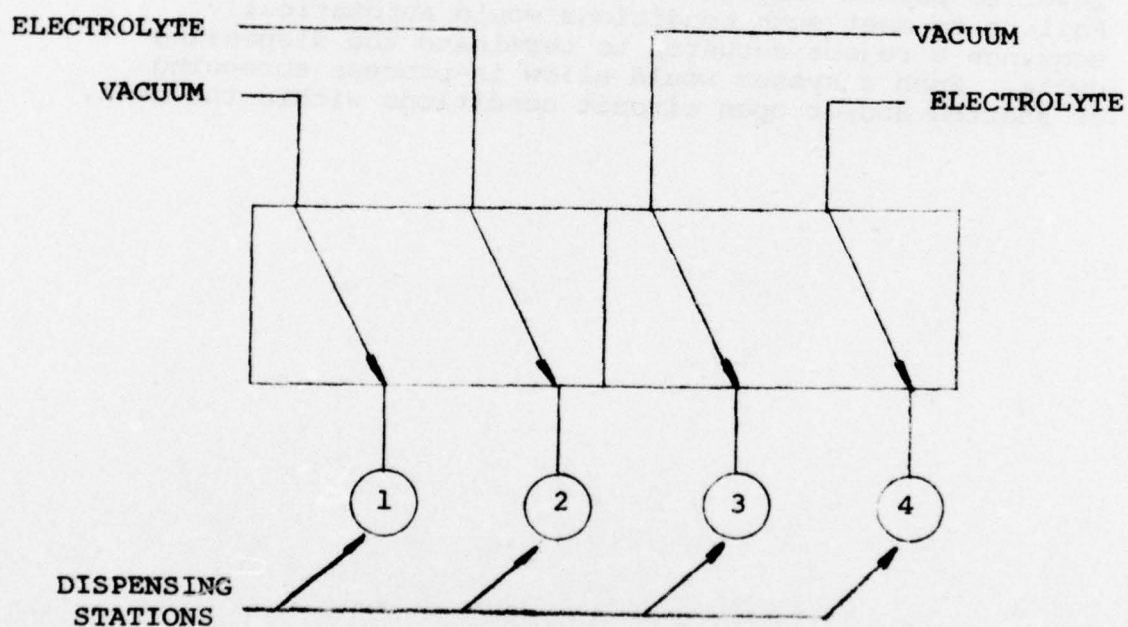
Same valve in the actu-
ated condition

ELECTROLYTE DISPENSER SHUTTLE VALVE

FIGURE 10

One proposed valve system as shown in Figure 11 will permit multiple evacuation and dispensing of electrolyte by actuation of the shuttle valves. Various valve component and seal materials are presently being considered to assure minimum equipment maintenance; especially those components which are continuously exposed to electrolyte. A four station dispensing system is currently proposed to attain the required production rate.

PCI is also considering the employment of an impedance measuring unit which would continuously monitor cell impedance or capacitance prior to the electrolyte dispensing cycle. This unit would be permanently mounted to the dispensing station. Cell impedance would be required to conform to a pre-determined acceptance level to permit completion of the dispensing cycle. Failure to meet such conditions would automatically sequence a reject actuator to terminate the dispensing cycle. Such a system would allow in-process screening of shorted and/or open circuit conditions within the cell.



ELECTROLYTE DISPENSING SCHEMATIC

FIGURE 11

VIII. HERMETIC SEAL AND CELL CLOSURE

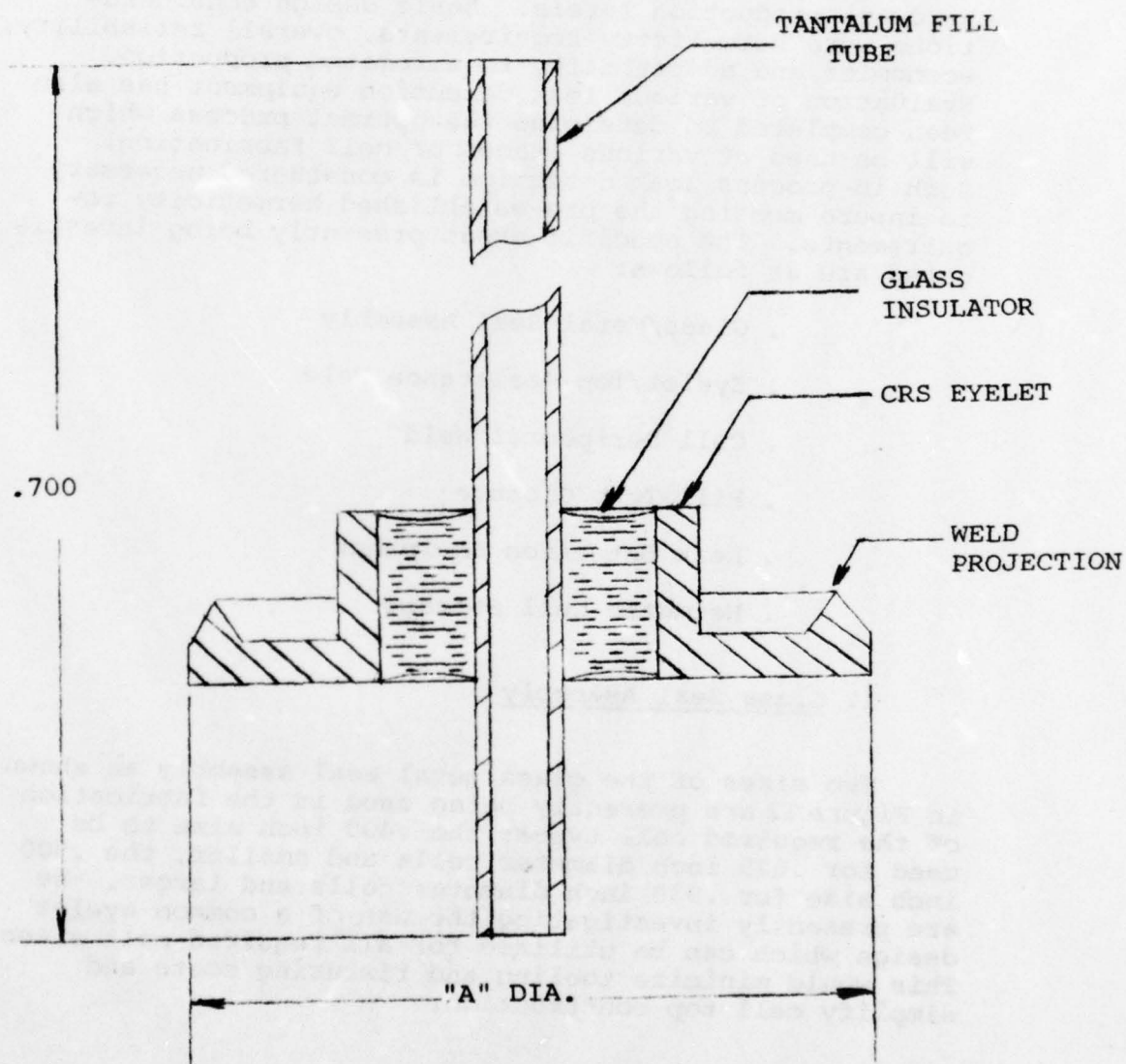
During the second quarterly period, significant effort has been directed toward finalizing a hermetic cell top design and demonstrating the closure techniques which will be used to meet the required hardware production levels. Basic design considerations were hermeticity requirements, overall reliability, economics and adaptability to automated production. Evaluation of various leak detection equipment has also been completed to determine the optimal process which will be used at various stages of cell fabrication. Such in-process leak detection is considered necessary to insure meeting the pre-established hermeticity requirements. The specific areas presently being investigated are as follows:

- . Glass/Metal Seal Assembly
- . Eyelet/Top Resistance Weld
- . Cell Peripheral Weld
- . Fill Tube Closure
- . Leak Detection Equipment
- . Hermetic Cell Storage

1. Glass Seal Assembly

Two sizes of the glass/metal seal assembly as shown in Figure 12 are presently being used in the fabrication of the required cell types; the .400 inch size to be used for .625 inch diameter cells and smaller, the .500 inch size for .930 inch diameter cells and larger. We are presently investigating the use of a common eyelet design which can be utilized for all required cell sizes. This would minimize tooling and fixturing costs and simplify cell top construction.

The seal primarily consists of a tantalum fill tube, a steel eyelet and a glass insulator preform; the assembly of which is thermally fused to effect an hermetic compression seal. A detailed engineering specification for the glass/metal seal assembly has been developed to quantitatively define the following physical,



"A" DIA.
 -1 .500 INCH
 -2 .400 INCH

GLASS/METAL SEAL ASSY.

FIGURE 12.

electrical, hermeticity and environmental requirements:

- . Visual Inspection
 - Dimensional configuration
 - Evidence of cracks, voids, delamination
 - Surface cleanliness
- . Hermeticity Test @ 2.0×10^{-8} atm cc/sec maximum leak rate
- . Thermal Shock @ -65 to 125°C (3 cycles)
- . Dielectric withstanding voltage @ 1500 volts AC
- . Insulation resistance @ 500 volts DC, 50 megohms minimum
- . Pressure Test @ 500 psi for 30 seconds minimum

The above parameters will be continuously monitored and tested by the glass seal manufacturer to assure product uniformity and reliability. Certificates of material compliance will also be submitted in addition to the above test results.

Effort is continuing to formulate a cleaning process to remove surface oxidation from the tantalum tube which is formed during the glass fusion process. Such oxidation prevents successful spot welding of the aluminum cathode tab to the tantalum fill tube. It was found that an increased electroless nickel plating thickness is required to prevent eyelet damage during the cleaning process. Various solvents are presently being evaluated to assure complete removal of any residual acid which may degrade the glass seal.

Tantalum inter-connection tab material has also been procured and will be evaluated as an interim solution. Voltage drops across the welded joint will be measured at various current levels to determine the feasibility of this design change.

2. Eyelet/Top Resistance Weld

Numerous samples of each size glass seal assembly were resistance welded to various size top shells using a 60 KVA resistance welder equipped with a low inertia head to provide quick response at constant pressure during the entire weld cycle. The units were subsequently inspected and tested to quantitatively measure the leakage rate using a helium mass spectrometer. Maximum leak rate observed was 2×10^{-8} cc/sec helium which is well within the specified limits of the contract.

The units were also subjected to a pressurized pneumatic bubble test. Each welded assembly was exposed to 160 psi of dry air while immersed in an alcohol solution for up to 24 hours. No evidence of leakage was observed on any of the tested samples.

Resistance weld parameters are currently being finalized to insure consistent welds which meet or exceed all structural and hermeticity requirements.

3. Cell Peripheral Weld

a. Plasma Arc Weld

Hermetic closure of the cell periphery is presently being accomplished using a plasma arc welding process. One problem associated with the peripheral weld has been weldment porosity at the joint overlap. This condition is primarily due to rapid cooling of the joint interface at the termination of the weld cycle. This tends to produce a structurally weak joint which is porous, especially during exposure to high pressure. This discrepant condition has now been corrected by incorporation of a down slope function as part of the weld cycle. This operation gradually reduces the weld current levels at the termination of the weld cycle and results in slow cooling of the joint thereby eliminating weldment porosity at the overlap.

Another problem encountered during the peripheral weld was the flow characteristics of the molten metal

at the joint interface. High weld currents were previously required to overcome the surface tension of the molten metal to produce a uniform and consistent peripheral bead. However, such weld current levels cause excessive heat generation which can degrade the heat sensitive components within the cell; especially the insulators, electrode separator and glass seal assembly. This problem has now been corrected by using a shielding gas containing 95% argon and 5% hydrogen. Such a mixture improves the flow characteristics and has permitted the use of lower weld current levels to achieve a hermetic peripheral weld on cell sizes ranging from .625 inch to 1.625 inch diameter.

Heat sinks and holding nests have also been fabricated for various size cells to transfer excess thermal energy away from the cell during the weld cycle to avoid degradation of heat sensitive components.

Implementation of the above corrective action has now resulted in a significant decrease in peripheral weld rejection rate.

Leak detection of the peripheral weld is qualitatively performed using a pressurized pneumatic bubble test. A maximum pressure differential of 160 psi of dry air is applied within the cell while immersed in an alcohol solution for 30 seconds. Alternative leak detection techniques are presently being evaluated in an effort to quantitatively measure leak rate within a minimum cycle time.

b. Laser Welder

Evaluation of various continuous wave neobium YAG laser welders has resulted in the following conclusions:

- . The YAG laser welder has insufficient power to effect a peripheral weld within the required time allotment; especially for large cell diameters. Efforts to increase weld speed resulted in inadequate beam penetration and poor structural weld characteristics

- . Excessive heat was developed during the long peripheral weld cycles which degraded heat sensitive components within the cell.
- . Operational cost and periodic maintenance requirements were not acceptable within a production environment.

Significant effort has now been directed toward achieving a hermetic peripheral weld using a CO₂ laser welder. The CO₂ laser as shown in Figure 13 essentially converts energy from an electrical discharge into molecular vibrational energy within the CO₂ gas. Photons are generated as some of the CO₂ vibrational energy is released. Alignment mirrors direct and focus these randomly emitted photons to sustain the photon generation, i.e. the lasing action.

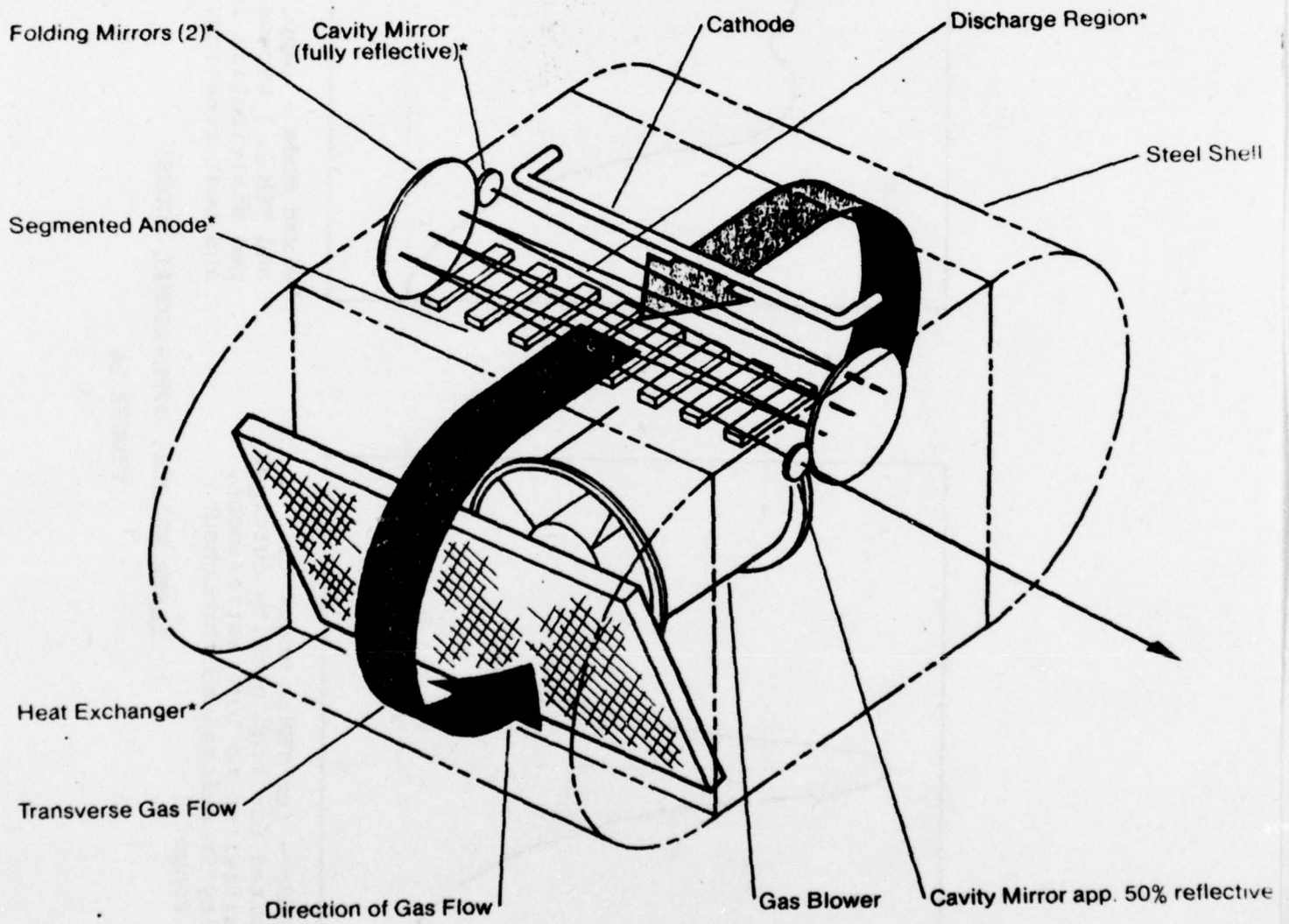
The CO₂ laser welder is available in several high power configurations which have been evaluated for peripheral welding of the various size cell cans. The results are as follows:

Weld Power (Watts)	Number Heads	Weld Rate inch/sec	Output/8 Hours* ("D" Cell)
250	1	0.5	2400
500	1	1.0	3600
500	2	0.5	4800
1000	1	4.0	5760
1000	2	4.0	11,520

*Assumes material handling (load/unload) time of 4 sec.

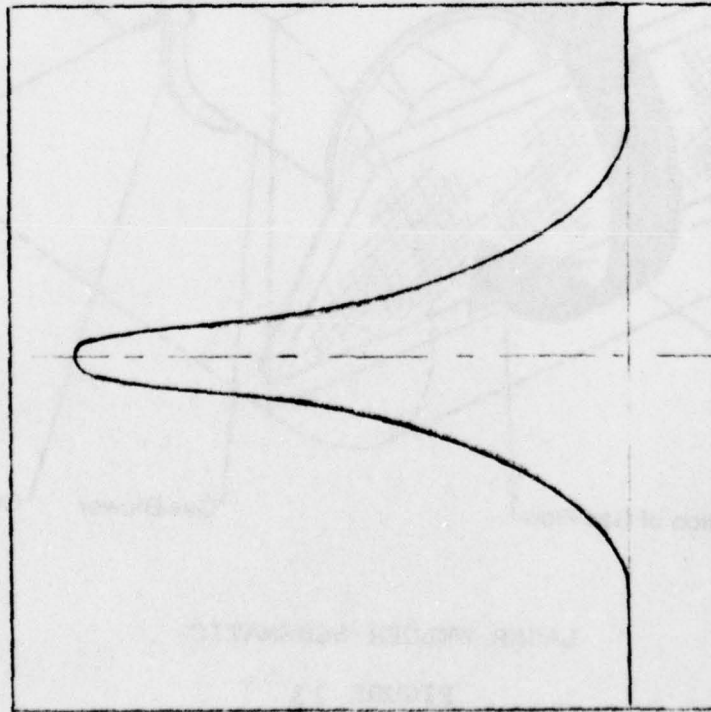
A two station (dual head or split beam) laser welder appears to be the most advantageous system since it minimizes "dead" time during in-process loading and unloading of cell sub-assemblies. Prototype samples using the above equipment are presently being fabricated and evaluated for hermeticity and structural integrity.

The CO₂ laser welder can be operated under two modes; a pulsed (Gaussian mode) and a continuous wave (mixed mode) as shown in Figure 14. Gaussian pulsed mode consists



LASER WELDER SCHEMATIC

FIGURE 13



Gaussian Mode - (or TEM₀₀ Mode) is ideally suited for high quality cutting. Power densities up to 10+9 watts/inch². Mode quality is maintained throughout the power range.



Mixed Mode - (app. 50% each of TEM₀₀ and TEM₀₁) because of its flat topped distribution is ideal for welding and heat treating.

LASER WELDER, OPERATIONAL MODES

FIGURE 14

of overlapping spots which concentrate high energy pulses over a prescribed duty cycle which minimized heat transfer to the workpiece. The mixed mode is a continuous welding cycle at a higher average energy level which permits faster weld speeds and greater throughput. Evaluations are currently being performed to determine the thermal characteristics of each mode and their effects on heat sensitive components within the cell.

c. Resistance Weld

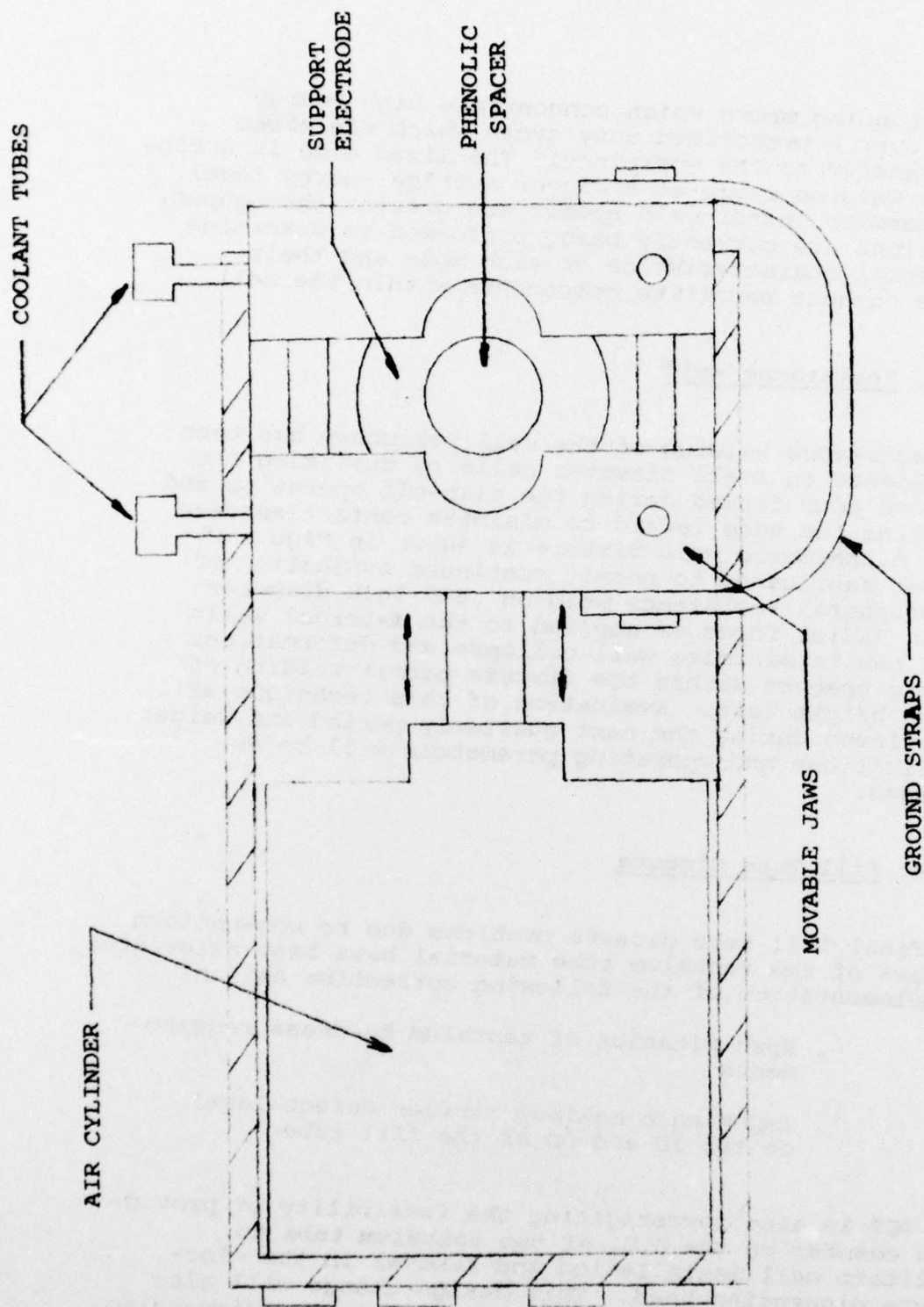
Resistance welding of the cell periphery has been accomplished on small diameter cells by utilizing the blunt can edge formed during the clip-off operation and by rolling the edge inward to minimize contact surface area. A pneumatic weld fixture as shown in Figure 15 has been fabricated to permit continued evaluation of the peripheral resistance weld on .625 inch diameter cells. Radial force is applied to the external walls of the can to minimize wall collapse and deformation. Phenolic spacers within the fixture permit welding of various height cans. Evaluation of this technique will be continued during the next quarterly period and welder specifications and operating parameters will be established.

4. Fill Tube Closure

Final fill tube closure problems due to non-uniform hardness of the tantalum tube material have been alleviated by implementation of the following correction action:

- . Specification of tantalum hardness requirements
- . Imposing a maximum surface defect level on the ID and OD of the fill tube

PCI is also investigating the feasibility of providing a chamfer on the O.D. of the tantalum tube to facilitate cell installation and removal in the electrolyte dispensing head. This design change will also minimize damage to the O-ring seals within the dispensing head and reduce the frequency of periodic seal replacement.



PERIPHERAL RESISTANCE WELD FIXTURE

FIGURE 15

5. Leak Detection Equipment

Various helium mass spectrometers have been evaluated for leak detection of the following:

- . Glass to metal seal assembly
- . Seal/Top assembly
- . Top/Can peripheral weld
- . Fill tube closure

Leak rate measurements of the above sub-assemblies will be continuously monitored throughout the fabrication of the hermetic cells to assure conformance to the hermeticity requirements which have been established at 2.0×10^{-8} atm cc/sec helium. Such leakage measurements will permit in-process screening of defective cell components and sub-assemblies. The helium mass spectrometer has a sensitivity of 1.0×10^{-10} atm cc/sec helium and is therefore well suited for this application.

We are also investigating the feasibility of introducing a trace amount of helium into the cell during the electrolyte dispensing operation. This will permit quantitative leak detection of the completed cell to verify hermeticity of the fill tube closure and will permit leakage measurements after cell exposure to elevated temperature storage environments.

6. Hermetic Cell Storage Program

Approximately one hundred fifty (150) hermetically sealed 660-5AS (1.625 inch diameter, 5.500 inch height) are presently undergoing a long term elevated temperature storage program. The cells are equipped with a bottom vent configuration and contain a tantalum tube glass seal assembly. Both test cells and control samples are being stored under various thermal environments in an effort to quantitatively measure the following:

- . Electrolyte leakage rates
- . Capacity discharge characteristics
- . Electro-chemical corrosion of the glass seal assembly

Results to date indicate no loss of cell weight after 30 days storage at 160°F. Capacity levels of these cells which were observed to be within normal distribution limits are as follows:

Storage Time (days)	Storage Temp. (°F)	Discharge Temp. (°F)	Load (ohms)	Service Life (2.0 volts) (avg. hours)
0	-	+70	1.9	17.5
0	-	-20	1.9	9.7
30	RT	+70	1.9	16.1
30	RT	-20	1.9	9.1
30	160	+70	1.9	15.8
30	160	-20	1.9	10.2

The tests will be continued for a period of six (6) months to obtain sufficient data verification to permit detection of any design problems.

IX. Conclusions

During the present quarter, effort has continued in accordance with the planned engineering objectives as defined in the revised PERT/TIME Network. These objectives include finalizing the production fabrication techniques and equipment design and subsequent implementation of such equipment in the construction of the required hermetic cells and batteries.

Hermetic cell and battery design has been re-evaluated in an effort to attain required cell capacity at minimum current density levels. An evaluation is also underway to determine the effects of various PCI electrolyte formulations on cell discharge performance and operation of the safety vent mechanism. Initial design and fabrication of a coined safety vent for small diameter cells is presently underway. The proposed design will occupy a minimal amount of internal cell volume and will therefore not reduce anticipated cell capacity. Preliminary tests indicate that the proposed side vent structure can be utilized on all cell sizes at a pressure of 425 psi.

Finalization of the anode fabrication equipment has proceeded to the design stages for integrating the anode tab cold welding technique and lithium rotary slitter design into a prototype production machine. Material transfer and alignment is presently being considered to minimize distortion of the electrode during the slitting process. A continuous feed of lithium and tab material appears to be the most feasible approach to automating this operation.

Initial fabrication of a continuous horizontal belt cathode machine is underway. Preliminary compression, sizing and drying of the slurry mix is presently under evaluation in an effort to determine the final design parameters. Sizing of the cathode thickness must be carefully controlled to avoid damage to the pore structure and alter porosity characteristics.

Two approaches for electrolyte dispensing are presently being considered; a pressure equilibrium system and a volumetric fill system. Various shuttle valves and metering systems are being considered in an effort to optimize electrolyte transfer and dispensing to meet the cell design specifications and tolerances. Incorporation of an impedance monitoring sensor may be required to prevent electrolyte dispensing within shorted cells.

Fabrication of the semi-automatic core winder is scheduled for completion in January 1977. Plans are already underway to

ascertain those modifications necessary to permit core winding of multi width/length electrodes using both non-woven and micro-porous polypropylene separators. Use of static eliminators may be required to prevent contamination of the separator surface.

Various welding techniques and equipment have been evaluated to accomplish hermetic closure of the cell components. A detailed engineering specification has been developed to quantitatively define all critical physical, electrical, hermeticity and environmental requirements. Refinement of the plasma arc welding process has been particularly successful; hermetic closure of cell cans ranging from .625 to 1.625 inch diameter has been reproducibly demonstrated. Both CO₂ laser welding and resistance welding equipment have been evaluated for accomplishing the peripheral weld. Final system selection will be made during the next quarterly period.

X. PROGRAM FOR 3RD QUARTER

The proposed program for the next reporting period will include the following:

- . Continued procurement of cell and battery prototype hardware and tooling.
- . Fabrication and evaluation of prototype hermetic cell assemblies.
- . Initial evaluation of an operational core winder and continuous cathode fabrication prototype machine.
- . Peripheral weld system selection and procurement of required components, fixtures and material handling equipment.
- . Re-evaluation of equipment design status and initial procurement and fabrication of prototype components.

XI. IDENTIFICATION OF PERSONNEL

The following additional personnel are presently involved in the subject program:

Thomas M. Watson

Education: B.S.E.E., Manhattan College, May 1969
Graduate Courses, New York University,
C.W. Post, 1970-73

Professional Experience:

Power Conversion, Inc.
Mt. Vernon, NY 10550

1976 - Present: Engineering Manager

Responsibilities include:

1. Management and supervision of all engineering functions for both research and production battery programs including the development of hermetic seal and safety vent techniques and cell optimization studies.
2. Establishment and surveillance of an automated production facility to permit expansion of existing prototype product lines for the Lithium/SO₂ system.
3. Preparation and review of technical and cost proposals for new development projects sponsored by various military and consumer organizations.
4. Management of configuration control and related documentation for hardware design, manufacturing tooling and equipment and the design and packaging of lithium batteries for specialized applications.

P.R. Mallory Corp.
Tarrytown, NY 10591

1973 - 1976: Senior Project Engineer, Lithium Power Systems

Responsibilities included:

1. Management and supervision of lithium battery prototype development programs.
2. Design of consumer and military power systems including the development of required performance and safety specifications.

3. Quality control surveillance of all production assembly and testing operations.
4. Preparation of program and cost proposals, product improvement presentations and customer liaison.

1969 - 1973: Grumman Aerospace Corp., Bethpage, NY 11714

Cognizant Engineer, Guidance Systems, Lunar
Module Program

Responsibilities included:

1. Establishment of all performance specifications for inertial guidance systems.
2. Engineering surveillance and inspection of inertial hardware assembly procedures and facilities.
3. Failure analysis and implementation of required corrective action and configuration control.
4. Evaluation of program costs, product improvements and customer liaison.

The labor hours expended during this quarterly reporting period are as follows:

<u>Dr. Stewart M. Chodosh</u>	166 hrs
Contracts Administrator and Program Manager	
<u>Martin G. Rosansky</u>	405 hrs
Senior Engineer	
<u>Thomas M. Watson</u>	405 hrs
Senior Engineer	
<u>Anandaram Joshi</u>	219 hrs
Test Engineer	
<u>Julius Cirin</u>	262 hrs
Technician	
<u>N. Bartilucci</u>	269 hrs
Technician	
<u>James Harris</u>	415 hrs
Technician	
<u>Prakash Jog</u>	346 hrs
Engineer	
<u>James Maguire</u>	323 hrs
Supervisor	

XII PUBLICATIONS AND REPORTS

No publications or reports were issued during this quarterly period.

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Institute for Defense Analyses ATTN: Mr. Robert Hamilton P. O. Box 55 Libertytown, MD 21762	1
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Commander US Army Materiel Development and Readiness Command ATTN: DRCRD-TC 5001 Eisenhower Avenue Alexandria, VA 22333	1
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Pentagon
Washington, DC 20301

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Mr. Richard E. Oderwald
Department of the Navy
Hqs, US Marine Corps
Code LMC 4
Washington, DC 20380

1

